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# TiB whiskers reinforced high temperature titanium Ti60 alloy composites with novel network microstructure

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ABSTRACT

TiB whiskers reinforced high temperature titanium Ti60 alloy (TiBw/Ti60) composites with a novel network microstructure have been successfully fabricated by the system of large spherical Ti60 powders and fine $TiB_2$ powders. The results show that the minimum temperature fabricating the composites by reaction hot pressing (RHP) is established to be 1300 °C due to usage of high temperature titanium Ti60 alloy. The TiB whiskers are in situ synthesized around large Ti60 matrix particles and then formed a novel three-dimensional (3D) network microstructure. The tensile strength of 8 vol.% TiBw/Ti60 composites with a network microstructure is increased by 61.1%, 57.4% and 45.5% compared with that of the monolithic Ti60 alloy at 600 °C, 700 °C and 800 °C, respectively. The superior improvement can be mainly attributed to the network microstructure, grain refinement and usage of large spherical Ti60 powders.


## 1. Introduction

In order to further enhance the mechanical properties of titanium based materials, much attention has been paid on titanium matrix composites (TMCs) due to its superior properties such as high specific strength, high specific modulus and high temperature durability [1–3]. In particular, discontinuously reinforced titanium matrix composites (DRTMCs), fabricated by in situ methods are sought-after due to their superior and isotropic properties along with low cost [4–6]. DRTMCs is most probably used as high temperature components in the field of aerospace, military and automotive. Therefore, the critical problem facing DRTMCs is improving the high temperature mechanical properties. Traditionally, researchers have always been seeking better reinforcements, better matrix alloys, better fabrication methods and more homogenous reinforcement distribution to improve the performance of DRTMCs [1]. It is unanimous that TiB whisker (TiBw) was regarded as the optimal reinforcement in Ti matrix [1]. Powder metallurgy (PM) coupled with in situ reaction synthesis such as reaction hot pressing (RHP) has been considered as an effective method to fabricate DRTMCs, largely due to its ability for microstructure control, near net shape processing and minimal material waste [1,7]. However, DRTMCs fabricated by the conventional PM technique exhibit extreme brittleness and only a limited improvement in performance [1,7].

Ti-1100 (USA), IMI834 (UK), BT39 (Russian) and Ti60 (China) alloys possessing the highest service temperature of 600 °C can be used as matrix to fabricate the highest temperature TMCs. As early as 1990s, high temperature titanium alloy Ti-1100 [8,9] and IMI834 [10] are used to fabricated continuous SiC fiber reinforced titanium matrix composites. Recently, Xiao et al. fabricated (TiBw + $La_2O_3$)/IMI834 composites [11] and (TiBw + TiCp + $La_2O_3$)/IMI834 composites [12] by melting technique, and obtained a superior high temperature creep resistance. Ti-1100 alloy was also selected to fabricated DRTMCs such as TiCp/Ti-1100 composites [13] and (TiBw + TiCp)/Ti-1100 composites [14]. By employing Ti6242 alloy, Lu et al. [15] have successful prepared in situ (TiBw + TiCp)/Ti6242 composites by melting technique followed by forging processing, and the ultimate tensile strength was improved to 639 MPa at 650 °C. In addition, Liu et al. [16,17] fabricated 10 vol.% TiCp/TA15 composites by laser melting deposition. The composites exhibited 625 MPa, 476 MPa and 342 MPa at 600 °C, 650 °C and 700 °C, respectively. However, there is no effort to fabricate high temperature titanium alloy matrix composites using Ti60 as matrix or using PM process.

In the past 40 years, the aim has been always to achieve a homogeneous microstructure of DRTMCs [1,7]. It is worth mentioning that both the strengthening effect and the toughening effect of DRTMCs were effectively improved by tailoring a novel network microstructure compared with a homogeneous microstructure in our previous work [2,18]. Moreover, the maximum service temperature of TiBw/Ti6Al4V composites was significantly increased by 150–200 °C on the basis of the same tensile strength [18].

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Therefore, it is significant and necessary to design and fabricate novel TiBw reinforced high temperature titanium Ti60 alloy composites with a novel network microstructure fabricated by PM process. This novel composite will exhibit the highest strength at high temperatures due to the employment of the best TiBw reinforcement, the highest temperature titanium Ti60 alloys as matrix, the most effective fabrication method (PM) and the novel network microstructure.

## 2. Experimental procedures

In order to fabricate TiBw/Ti60 composites with a network microstructure, the large and spherical Ti60 powders with a particle size of 120–220 μm (Fig. 1a), fine and prismatic $TiB_2$ powders with that of 1–8 μm (Fig. 1b) were selected in the present study. The analyzed composition (in wt.%) of Ti60 alloy is 5.9 Al, 4.2Sn, 3.5Zr, 0.4Mo, 0.38Nb, 0.93Ta, 0.38Si, 0.04Fe, 0.06C, 0.01N, 0.003H, 0.07O, 0.3 others, and balance titanium. The selected two powders were low energy milled at 200 rpm for 8 h with a milled media to material ratio of 5:1 under an argon atmosphere. The aim of low-energy milling process was not to break down the large Ti60 powders but to make fine $TiB_2$ powders be tapped onto the surface of the large Ti60 particles. Then, the mixed powders were hot pressed in vacuum ($10^{-2}$ Pa) at 1200 °C and 1300 °C under a pressure of 20 MPa for 60 min. TiB whiskers were synthesized during the reaction hot pressing process between Ti and $TiB_2$ according to the following Eq. (1), and then 5 vol.%, 8 vol.% and 12 vol.% TiBw/Ti60 composites with a network microstructure were fabricated.

$$Ti + TiB_2 \rightarrow 2TiB \tag{1}$$

Tensile specimens have gauge dimensions of 15 mm × 5 mm × 2 mm and a total of five samples were tested for each composite. High temperature tensile tests, which refers to the metal materials testing standards of ISO 783:1999 [19], were carried out in air using an Instron-1186 universal testing machine at 600 °C, 700 °C and 800 °C, and a constant crosshead speed of 0.5 mm/min (corresponding strain rate is $5.5 \times 10^{-4}\ s^{-1}$). Microstructure observation was performed using a scanning electron microscopy (SEM, Hitachi S-4700).

## 3. Results and discussions

Fig. 2 shows the X-ray diffraction pattern of the as-sintered 8 vol.% TiBw/Ti60 composites fabricated at 1300 °C, indicating that only Ti and TiB phases exist in the composites and no $TiB_2$ phase is detected. Similar results are also obtained for other composites including the composites fabricated at 1200 °C and the composites with 12 vol.% TiBw reinforcement. This result demonstrates that the in situ reaction between Ti and $TiB_2$ is easily completed, which is consistent with the previous work [20,21].

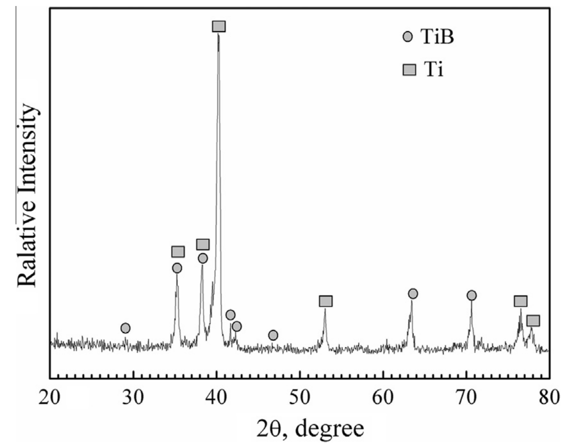


**Fig. 2.** X-ray diffraction pattern of 8 vol.% TiBw/Ti60 composites fabricated at 1300 °C.

Fig. 3 shows SEM micrographs of network structured 8 vol.% TiBw/Ti60 composites fabricated at 1200 °C and 1300 °C. As reported in the previous work [20,22], it is unanimous that TiB phase always displays whisker morphology due to its special B27 crystal structure. The reason is that the growth speed of TiBw along [0 1 0] direction is much higher than that along [1 0 0] and [0 0 1] directions. Combining with the XRD results, it is clearly that the TiBw reinforcement is in situ synthesized around Ti60 matrix particles, and then formed a 3D network microstructure. Therefore, the formation of network microstructure is mainly attributed to the usage of large spherical Ti60 particles, the low energy milling process and solid state sintering process (RHP).

However, it can be seen that many pores exist in the composites fabricated at 1200 °C, while the composites fabricated at 1300 °C are fully compacted. In the previous work [20], the TiBw/Ti6Al4V composites with a network microstructure can be fully compacted at 1200 °C. Therefore, in the solid sintering process, the compacting temperature of the composites is promoted due to usage of high temperature titanium Ti60 alloy possessing higher strength at high temperatures. It can be concluded that 1300 °C is the optimal sintering temperature in order to fabricate high temperature titanium alloy (such as Ti60, Ti1100 and IMI834 et al.) matrix composites with a novel network microstructure by RHP.

Fig. 4a shows the SEM micrograph of the as-sintered monolithic Ti60 alloy. It can be seen that the typical widmanstätten microstructure presenting large primary β grains and lamellar α phase is formed in the as-sintered Ti60 alloy. The size of the primary β

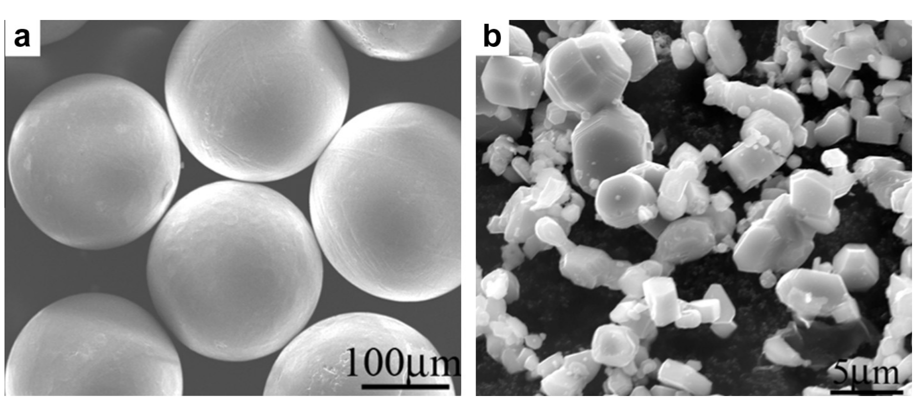


**Fig. 1.** SEM micrographs of raw materials: (a) Ti60 powders, and (b) $TiB_2$ powders.

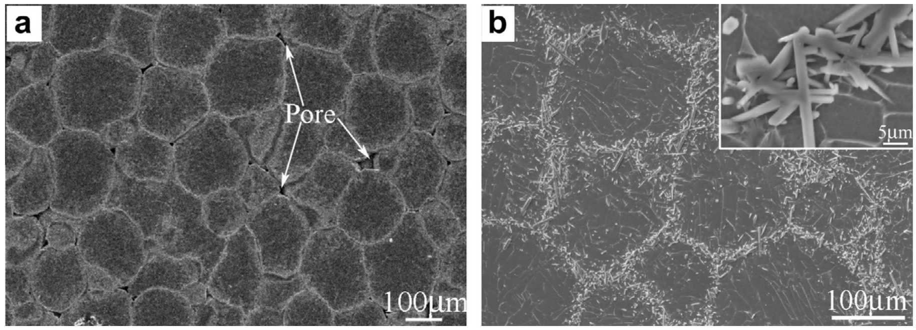


**Fig. 3.** SEM micrographs of the network structured 8 vol.% TiBw/Ti60 composites fabricated at (a) 1200 °C and (b) at 1300 °C.

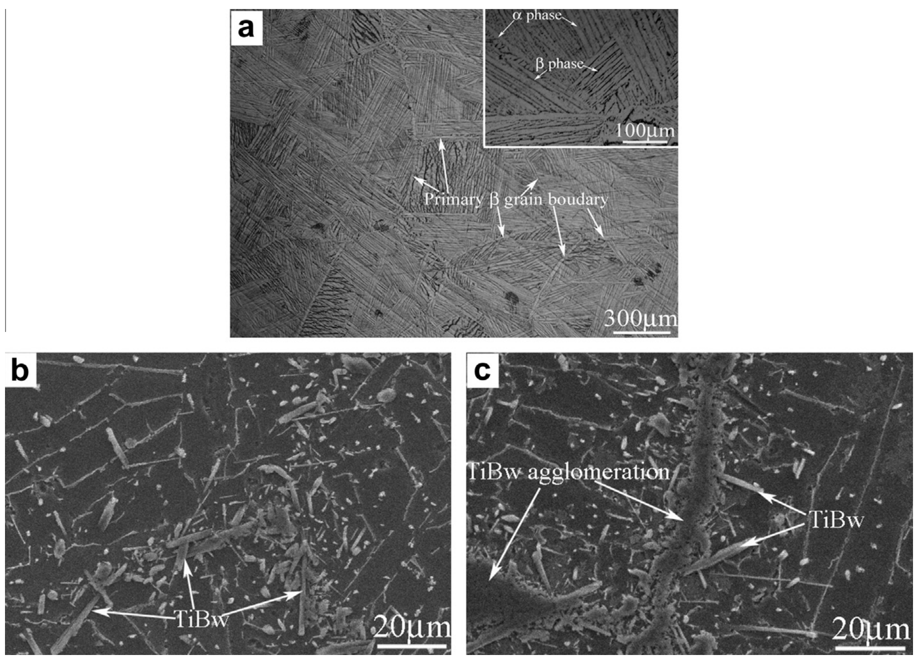


**Fig. 4.** SEM micrographs of (a) the monolithic Ti60 alloy, (b) 5 vol.% and (c) 12 vol.% TiBw/Ti60 composites with a network microstructure.

grains is much larger (~900 μm) than that of the as-received Ti60 powders, which is harmful to the mechanical property. However, the size of the primary β grains of the TiBw/Ti60 composites with a network microstructure is constrained to be similar with that (~200 μm) of the as-received Ti60 powders (Fig. 3), due to the existence of reinforcement with a network distribution. Additionally, as shown in Figs. 3b, 4b and c, the size of α phase in the composites is inhibited to 10–20 μm, which is much smaller than that (over 300 μm) in the unreinforced Ti60 alloy (Fig. 4a). This phenomenon can be attributed to the existence of network reinforcement, which was also observed in the TiCp/Ti6Al4V composites with a network microstructure [23]. These are equal to refinements of the β grain and α phase, which are beneficial to the mechanical property.

In the composites with a network microstructure, the TiB whiskers are homogenously distributed in the network boundary. In other words, ceramic TiB whiskers are introduced into the β grain boundary, which not only overcomes the weakening effect of grain boundary at high temperatures, but also increases the strengthening effect of grain boundary [18]. In addition, the TiB whisker is grown as dowel-like structure due to its B27 structure [1], which can effectively joint the adjacent Ti60 matrix particles (Fig. 4b and c). Moreover, the local volume fraction of TiBw in the network boundary increases with increasing the overall volume fractions as seen from Figs. 3b, 4b and c. For the 5 vol.% TiBw/Ti60 composites, the microstructure is bi-continuous embodying continuous matrix and quasi-continuous reinforcement (Fig. 4b), which is beneficial to the combination of mechanical properties. When the volume fraction is increased to 8 vol.%, the microstructure becomes quasi-continuous matrix and continuous reinforcement as seen from Fig. 3b. Moreover, mechanical-bonding and self-jointing structures except for single whisker can be clearly observed. These are positive to the strengthening effect of TiBw reinforcement. But, when the volume fraction to 12 vol.%, the structure of continuous TiBw agglomeration and discrete matrix particles replaces the above bi-continuous structure, which is certainly harmful to the mechanical property of the composites. The formation of TiBw agglomeration can be attributed to that Ti source for reaction synthesis is insufficient near local network boundary due to excessive $TiB_2$ addition.

Table 1 shows the room temperature tensile properties of the as-sintered TiBw/Ti60 composites and the as-sintered unreinforced Ti60 alloy. The tensile strength of the 5 vol.% and 8 vol.% TiBw/Ti60 composites is increased from 1010 MPa to 1160 MPa and 1180 MPa, coupled with a remarkable decrease of tensile elongation from 4.5% to 3.3% and 1.6%, respectively. These can be attrib-

**Table 1**
Comparative study of the room temperature tensile properties of TiBw/Ti60 composites and Ti60 alloy.

| Properties | Ti60 | 5 vol.% | 8 vol.% | 12 vol.% |
|---|---|---|---|---|
| $\sigma_b$ (MPa) | 1010 ± 7 | 1160 ± 8 | 1180 ± 10 | 990 ± 10 |
| $\delta$ (%) | 4.5 ± 0.3 | 3.3 ± 0.2 | 1.6 ± 0.2 | – |

uted to the increasing reinforcement contiguity and the decreasing matrix contiguity (Figs. 3b and 4b). Moreover, the 12 vol.% TiBw/Ti60 composite exhibits brittle fracture without elongation due to the totally continuous TiBw ceramic network boundary (Fig. 4c). Therefore, the strength is drastically decreased to 990 MPa because of its brittle fracture.

Fig. 5 shows the high temperature tensile properties of the monolithic Ti60 alloy and the network structured 5 vol.%, 8 vol.% and 12 vol.% TiBw/Ti60 composites. The tensile strength of the composites increases with increasing the TiBw volume fractions at high temperatures, while that of 12 vol.% TiBw/Ti60 composites falls down due to the continuous TiBw agglomeration (Fig. 4c). For the 5 vol.% TiBw/Ti60 composites, the tensile strength is increased to 787 MPa, 625 MPa and 396 MPa from 552 MPa, 458 MPa and 303 MPa at 600 °C, 700 °C and 800 °C, respectively. Furthermore, the tensile strength of 8 vol.% TiBw/Ti60 composites is increased to 889 MPa, 721 MPa and 453 MPa, respectively. That is to say, the tensile strength can be increased by 61.1%, 57.4% and 45.5% compared with that of the monolithic Ti60 alloy at 600 °C, 700 °C and 800 °C, respectively. It is worth pointing out that the tensile strength of the composites is certainly further increased by subsequent heat treatment according to the previous experience [24]. Compared with the tensile strength of 625 MPa and 342 MPa at 600 °C and 700 °C of 10 vol.% TiCp/TA15 composites [16], the present composites exhibit an obvious improvement in high temperature tensile strength. Moreover, the strength of 721 MPa at 700 °C of the present 8 vol.% TiBw/Ti60 composite is also higher than that of 639 MPa at 650 °C of 8 vol.% (TiBw + TiCp)/Ti6242 composites [15]. Therefore, the high temperature tensile properties or the service temperature of DRTMCs are enhanced by fabricating TiBw/Ti60 composites with a network microstructure. The superior improvement in high temperature strength can be interpreted as follows: TiB whiskers with dowel-like structure as ceramic reinforcement are introduced into the grain boundary (network boundary) and formed into the novel network microstructure. This can effectively decrease the grain boundary weakening effect at high temperatures. Moreover, the in situ synthesized TiBw can exhibit the effective grain boundary strengthening effect even at high temperatures. Particularly for the 8 vol.% TiBw/Ti60 composites, the continuous TiBw network boundary can dominate the tensile behavior of the composites (Fig. 3b). In addition, the mechanical-bonding and self-jointing structures of TiB whiskers are certainly positive to the strengthening effect at high temperatures by increasing the reinforcement contiguity. However, the 12 vol.% TiBw/Ti60 composites just exhibit slight improvement even decrease in high temperature tensile strength. Moreover, the decreasing tendency is small with increasing temperatures. Both can be attributed to that the continuous TiBw agglomeration with a network microstructure dominates the tensile behavior of the composites.

In addition, the tensile elongation of the composites decreases with increasing TiBw volume fractions and increases with increasing temperatures. It is worth pointing out that the tensile elongation of 5 vol.% TiBw/Ti60 composites slightly decreases to 9.2%, 12.8% and 19.2% from 12%, 15.6% and 21.8% at 600 °C, 700 °C and 800 °C, respectively. Therefore, the 5 vol.% TiBw/Ti60 composites exhibit a superior combination of mechanical properties due to the dowel-like TiBw and the bi-continuous network microstructure (Fig. 4b). Even for the 8 vol.% TiBw/Ti60 composites with a network microstructure, the elongation keeps 7.5%, 9.2% and 11.7%, which can be viewed as a superior improvement considering the 61.1%, 57.4% and 45.5% improvement in high temperature tensile strength.

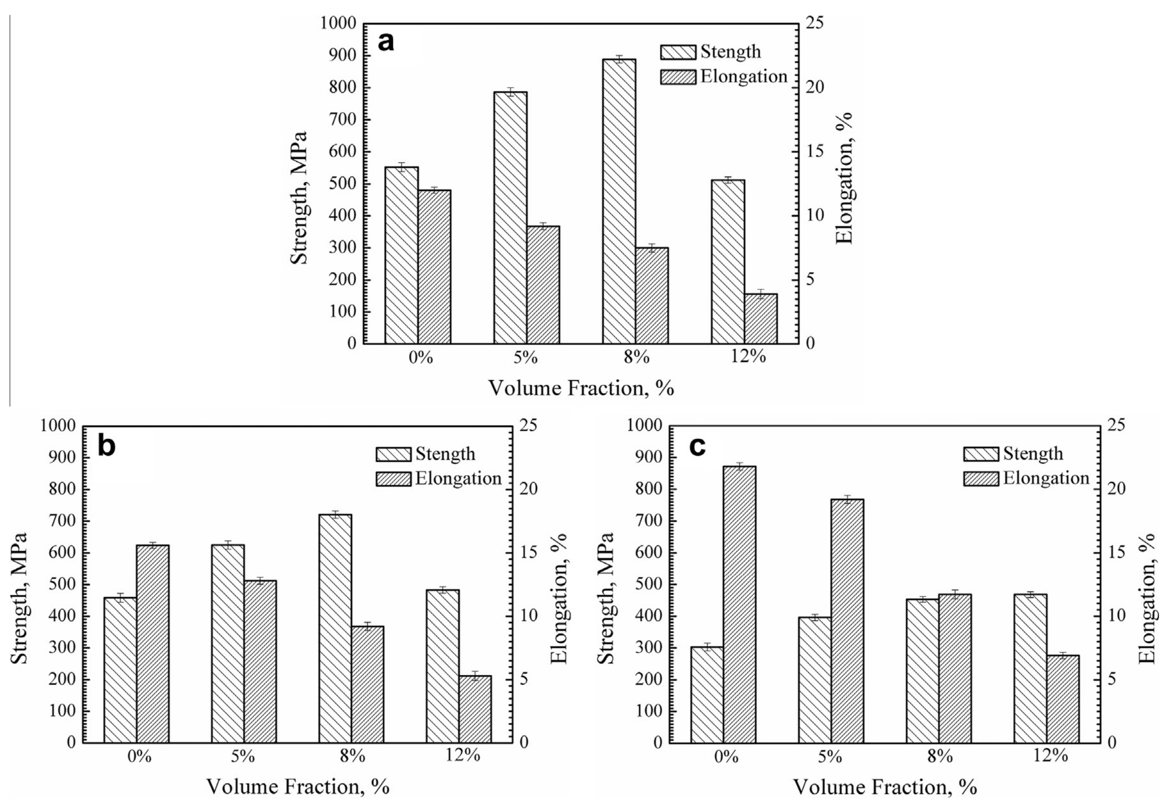


**Fig. 5.** Tensile properties of the monolithic Ti60 alloy and the network structured 5 vol.%, 8 vol.% and 12 vol.% TiBw/Ti60 composites at (a) 600 °C, (b) 700 °C and (c) 800 °C.

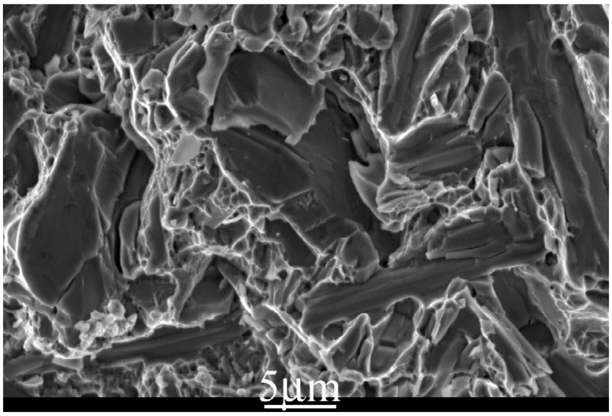


**Fig. 6.** Fracture morphology of 5 vol.% TiBw/Ti60 composite tested at 700 °C.

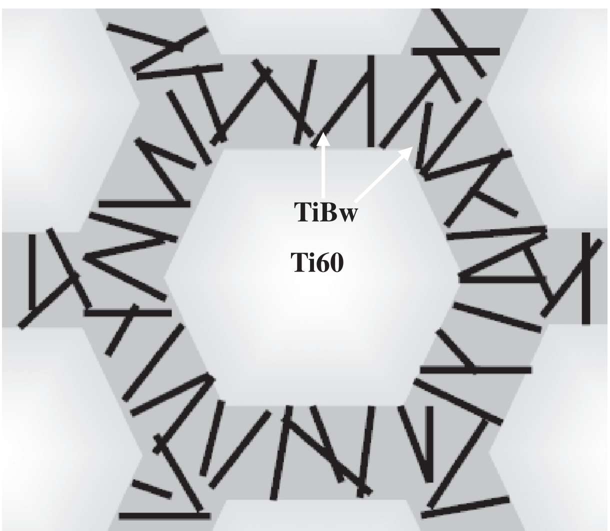


**Fig. 7.** Schematic illustration of TiBw/Ti60 composites with a novel network microstructure.

In summary, the maximum service temperature of the TiBw/Ti60 composites can be increased by over 100 °C while retaining the same strength of the monolithic Ti60 alloy, along with a suitable elongation. In addition, it is reasonable that both the strength and the elongation can be further increased by the subsequent deformation such as hot extrusion [25].

Fig. 6 shows the fracture surface of 5 vol.% TiBw/Ti60 composite tested at 700 °C. It is clearly seen that most of TiBw are fractured during the tensile process at high temperature, which indicates the effective strengthening effect of the TiBw reinforcement due to the strong interfacial bonding. Additionally, the dimples and tearing ridge lines around the TiBw reinforcement correspond to a superior tensile elongation of 12.8% at 700 °C (Fig. 5b). This can be attributed to the continuous matrix across the network boundary (Fig. 4b).

Fig. 7 shows the schematic illustration of TiBw/Ti60 composites with a novel network microstructure. As seen from Fig. 7, the TiB whiskers can effectively joint the adjacent Ti60 matrix particles like dowel connectors. The addition of TiBw ceramic reinforcement in the boundary can effectively overcome the grain boundary weakening effect at high temperatures. Moreover, the TiB whiskers reinforcement, particularly for the mechanical-bonding and self-jointing TiBw reinforcement can exhibit an effective grain boundary strengthening effect. In addition, the quasi-continuous TiBw network structure and the continuous TiBw network structure can inspire a superior strengthening effect by dominating the tensile behavior of the composites. It is important that the Ti60 alloy as matrix possessing high strength at high temperatures can support the strengthening effect of the network microstructure and TiBw reinforcement. In summary, the superior strengthening effect of TiBw/Ti60 composites with a network microstructure is described by the schematic illustration.

## 4. Conclusions

In order to further enhance the high temperature mechanical properties of DRTMCs, a high temperature titanium Ti60 alloy as matrix, the best TiBw as reinforcement, a novel network microstructure, an effective method of powder metallurgy (PM) coupled with in situ reaction synthesis were employed to fabricated the novel TiBw/Ti60 composites with a network microstructure. The work leads to the following findings:

(1) TiB whiskers reinforced high temperature titanium Ti60 alloy composites with a network microstructure were successfully fabricated by reaction hot pressing and using the large spherical Ti60 powders and fine $TiB_2$ powders.
(2) The optimal sintering temperature of TiBw/Ti60 composites with a network microstructure is established to be 1300 °C higher than that of TiBw/Ti64 composites.
(3) The tensile strength of 8 vol.% TiBw/Ti60 composites is increased by 61.1%, 57.4% and 45.5% compared with that of the monolithic Ti60 alloy at 600 °C, 700 °C and 800 °C, respectively.
(4) The superior improvement in high temperature tensile properties can be mainly attributed to the network microstructure, grain refinement, TiB whiskers and usage of large spherical Ti60 powders.

## Acknowledgements

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